TECHNICAL MEMO 5

SUMMARY AND EVALUATION OF INTERIM SOIL TEST MATERIAL SAMPLE ANALYSIS BY SEM AND IR

1.0 INTRODUCTION

USEPA Region 8 is currently engaged in a program to test and evaluate a variety of analytical methods for quantification of asbestos in site soils, vermiculite insulation, and other related site samples. As part of this program, an initial pilot study was performed using a set of "interim soil test materials" (ISTMs) with the aim of allowing a rapid initial assessment of the relative performance of infrared spectrometry (IR) and scanning electron microscopy (SEM) to quantify soil concentrations in the range of 0.1% to 1%. In addition, a set of Libby field samples with concentrations (estimated by PLM) ranging up to 5% asbestos were included for a qualitative comparison of PLM and IR/SEM results. This technical memo summarizes the approach and the results for these soil-based samples. A separate technical memo summarizes the results for a set of interim test materials prepared using ground quartz as the matrix.

2.0 ISTM PREPARATION

ISTM samples used in this pilot study consisted of two different types: soils spiked by USGS with known weight percents of Libby amphibole material, and authentic Libby field soil samples. These two sample groups are described below.

2.1 Spiked Samples Prepared by USGS

Spiked ISTM samples were prepared by USGS in concentrations ranging from 0.1 weight percent (%) up to a maximum of 0.8% asbestos using soil material collected from a site in the Libby, Montana area and a single soil sample collected from the Denver Federal Center (DFC) in Lakewood, Colorado. The collection, preparation and mixing of these materials is described below.

Amphibole Spiking Material

Amphibole material used to spike these soil matrices was obtained from a composite of ore samples collected from six locations at or near the Libby, Montana vermiculite mine site. The six samples

were selected for this work because USGS analysis of their mineralogical composition found that they were highly enriched in asbestos (about 80% pure), contained the full range of amphibole types found at the Libby site, and were relatively free of interfering contaminants.

Samples were initially air dried in their original plastic bags and, when necessary, disaggregated to reduce the particle size to less than 3 cm in diameter. The six samples were then ground in a Hardinger horizontal grinder equipped with 3-inch diameter steel plates, producing material with a particle size less than 2 mm in diameter. The ground material was transferred to a 10 gallon drum fitted with a customized expanded steel mixing baffle. The drum was sealed and transferred to a horizontal roller apparatus where the sample was allowed to mix for a total of 24 hours. After mixing, the drum was relocated into a HEPA hood, and samples were removed for the final grinding step. In this grinding step, 200 g of amphibole material, 1 L of deionized water, and 200 ceramic grinding cylinders (2 cm x 1 cm) were transferred to a 5 L ceramic ball mill and the mill was sealed. The ball mill was transferred to the horizontal roller apparatus and allowed to grind the sample for a total of four hours. After grinding, the ball mill was relocated to the HEPA hood and the ceramic cylinders were removed and rinsed with deionized water. The ground amphibole was transferred from the ball mill into a plastic drying tray and the aqueous suspension was allowed to evaporate over several days. Once dry, the amphibole material was disaggregated using a metal spatula and transferred to a series of wide mouth one-liter glass jars and then sealed. The composite sample was coarse ground using a three inch horizontal grinder equipped with steel plates. Approximately half the batch was later wet ground in a four liter ball mill using corundum grinding cylinders (1") to produce a fine grained aliquot. These coarse and fine grained amphibole spiking materials were used to prepared a variety of ISTMs, as described below.

DFC Soil Matrix ISTM Samples

Soil collected from the Denver Federal Center (DFC) was collected from a site chosen at random from an area located on the south east section of the center. During collection an area approximately one square meter in size was cleared of all surface debris and the top 3 inches of grass removed. Sufficient soil was removed to a depth of approximately eight inches to fill a five gallon plastic bucket (50 lbs). The soil was oven dried at room temperature in plastic lined cardboard trays (12x24x2 in). After drying the soil was processed through the USGS soil disaggregator and then passed over a 2 mm screen. Material passing the screen was collected for use in the soil test sample preparation. The soil material is dark brown in color, contains minor amount of fibrous organic material, and small pebbles (<2mm). The soil does not contain detectable levels of amphibole (<0.1%) as measured by XRD.

Approximately 2500 g of DFC soil was transferred to one gallon container fitted with a plastic mixing card. The soil sample was mixed for four hours and then split into 500 g aliquots using a standard Jones splitter. Each aliquot was transferred to a 1-liter wide mouth glass container. Aliquots of fine or coarse grained Libby amphibole material were added (HEPA hood) to each container in order to obtain amphibole concentrations of 0.1, 0.3, 0.6, 0.8 weight percent (%). Assuming the spiking material is about 80% pure, this corresponds to nominal concentrations of 0.08, 0.24, 0.48, and 0.64% asbestos. The amphibole aliquots were added to the soil as aqueous suspensions along with approximately 500 ml of deionized water. The container was sealed, vigorously shaken for approximately five minutes, uncapped, and then mixed using an overhead stirrer for approximately 1 hour. After mixing the slurry was quantitatively transferred to a 9 x 14 x 1 inch metal tray lined with aluminum foil. The tray was placed on a hot plate and allowed to dry over night at ~90C. The next day the sample was hand ground using a two liter ceramic mortar and pestle to disaggregate coarse particles. The mixture of coarse and fine grained soil material was returned to its original 1 liter glass bottle (cleaned and dried) which was fitted with a plastic mixing card. The container was sealed, transferred to a horizontal roller and mixed for approximately two hours. After mixing aliquots (~20g) were removed from the container using a sample thief and transferred to one ounce glass bottles. The bottles were labeled with a code identifying the soil used in the preparation, amphibole concentration, and texture of the amphibole (fine, coarse) used.

Libby Soil Matrix ISTM Samples

Soil material from the Libby, Montana area used in the preparation of these test samples was collected from four locations in the Libby area. The four samples were selected for use because the USGS found they had lower concentrations of massive amphibole than other soil samples from Libby. Fibrous amphibole was not detected by SEM examination. The samples were light brown in color, contained a minimal amount of visible fibrous organic material and were easily disaggregated.

The four soil samples were transferred to a new one gallon cardboard container fitted with a customized plastic mixing card. The container was covered, sealed with tape and then transferred to a horizontal roller apparatus where it was mixed for a total of six hours. After mixing the sample was split by hand into three 500 g aliquots in a HEPA hood. Each aliquot was transferred to a one liter wide mouth glass jar.

All soil used to prepared the Libby ISTM samples was prepared by passing the soil through a soil disaggregator and sieving through a 2 mm screen. To each aliquot of prepared soil, a separate amount of coarse ground Libby amphibole was added wet. A total of 200 ml of deionized water

was then added to the jar and the amphibole/soil mixture was mechanically mixed using a overhead stirred equipped with a customized stirrer. The sample was mixed for a total one hour. The sample was then quantitatively transferred to a 9 x 14 x 1 inch metal tray lined with aluminum foil. The tray was placed on a hot plate stirred to evenly distribute the mixture and then allowed to dry overnight at ~90C. The next day the dried sample was lightly ground using a 2 L ceramic mortar and pestle to disaggregate coarse particles. The sample was then transferred back into its original one liter wide mouth glass container which had been fitted with a plastic mixing card. The sample was then mixed for two hours using the horizontal roller. Individual samples (20g) were removed from the container using a sample thief and transferred to one ounce glass bottles. The bottles were labeled with a code identifying the soil used in the preparation, amphibole concentration, and texture of the amphibole (fine, coarse). Amphibole concentration in Libby soil samples had concentrations of 0.2, 0.65, 0.8 weight percent. Assuming the spiking material is about 80% pure, this corresponds to nominal concentrations of 0.16, 0.52, and 0.64% asbestos.

2.2 Unspiked Libby Field Samples

The ISTM data set also contained a number of authentic soil field samples from Libby. These were selected for inclusion in the pilot evaluation based on the PLM results for the samples. These samples are summarized below:

PLM Result	Number of samples
ND	4
Trace	3
Quant (1%-5%)	5

3.0 ANALYSIS

One set of 38 samples was sent to each of two laboratories: EMSL Analytical, Inc. (EMSL), and Reservoirs Environmental Analytical Services (RESI). These samples are summarized in Table 1. EMSL analyzed samples by infrared spectroscopy (IR) and by scanning electron microscopy (SEM), while RESI analyzed the samples by SEM. The SOP for SEM is provided as Attachment 1. The SOP for IR is currently proprietary, and will be made available at a later date. A brief description of each method is provided below.

SEM

The SEM method involves examination of multiple fields of view at a series of different magnifications. At each magnification, the analyst records the area fraction of the field that is occupied by asbestos structures within a specified size range. Following completion of the analysis, the mass fraction is estimated using an equation that combines the results across each of the magnifications, assuming that area fraction and mass fraction are equivalent.

IR

In the IR method, the sample is spread out in a petri dish, and the IR spectrum is recorded at multiple locations across the surface of the sample. The concentration (mass percent) is estimated from the average of the multiple readings, using a empiric calibration curve. Because the IR method does not distinguish between massive and fibrous amphibole, all samples that are positive by IR are examined by PLM to determine if the response is due to massive rather than fibrous amphibole. In addition, all samples that are negative by IR are also screened by PLM as a double check that no visible asbestos is present.

It should be noted that the results of these pilot studies are expected to lead to improvements in the SOPs for one or both methods, and that the SOPs for these methods will be revised in the future, as appropriate.

4.0 RESULTS AND DISCUSSION

4.1 Quantitative Analysis

Table 2 gives the raw analytical results for each sample. These results are discussed and evaluated below.

SEM Analysis

Figure 1 summarizes the SEM results from EMSL (upper panel) and RESI (lower panel) for the ISTM samples spiked with known amounts of asbestos by USGS. Note that the figures uses a log-log scale, so differences between expected and observed values tend to be compressed.

Inspection of Figure 1 reveals that both laboratories tended to underestimate the spiked mass percent. The underestimation tended to be less for DFC soils spiked with coarse amphibole

material, and greatest for DFC soil spiked with fine-ground material. The reason for the underestimation is not known.

Figure 2 summarizes the SEM results from EMSL and RESI for authentic field samples from Libby. The value plotted on the x-axis is the mass percent by PLM, which may not be an accurate reflection of the true concentration in all cases. For convenience, values reported as "ND" by PLM are plotted at an assumed concentration of 0.05%, and samples reported as "<1%" (Trace) by PLM are plotted at an assumed concentration of 0.5%. As seen, SEM tends to report substantially lower concentrations than PLM. The basis for this discrepancy is unknown, but could result either from a tendency to under-report by SEM and/or a tendency to over-report by PLM.

Figure 3 provides an inter-lab comparison of SEM results from EMSL and RESI. The correlation coefficient (R) and the coefficient of determination (R^2) are as shown below:

Sample Type	R	R^2
Spiked DFC (Coarse)	0.370	0.137
Spiked DFC (Fine)	0.993	0.986
Spiked Libby (Coarse)	-0.264	0.070
Unspiked Libby	0.886	0.785

As seen, correlation is relatively poor for samples spiked with coarse amphibole material. The correlation appears to be better for samples spiked with fine-ground material and for Libby field samples, but this is an artifact stemming from the fact that most of these samples were at or near the detection limits for both laboratories.

IR Analysis

Figure 4 presents the results of IR analysis by EMSL of the USGS spiked samples (upper panel) and the unspiked Libby field samples (lower panel). Note that the IR results are bounded between a lower reporting limit of 0.1% and an upper reporting limit of 1.0%. Non-detects are plotted below the lower reporting limit line, and values off-scale high are plotted above the upper reporting limit line.

As seen, although some results were close to expected, there is a general tendency for IR to underestimate the concentration of asbestos in both data sets. The basis of the underestimation is not known.

4.2 Semi-Quantitative Analysis

Basic Approach

It is important to realize that the current program of soil investigation at Libby does not necessarily require precise quantification of asbestos levels in soil to support decision-making. Rather, the current program at Libby is of a "screening" nature and seeks to classify each soil sample into one of three bins:

Bin	Concentration Range
A	< 0.1%
B	0.1%-0.9%
C	≥ 1%

In all cases, analytical results are rounded to the nearest 0.1% before assignment to bins.

There are four basic types of mis-classification errors that could occur in the application of the semi-quantitative system described above. However, not all are of equal concern. The types of errors and the impact on decision-making are described below.

False Positives

Case 1. The first type of mis-classification is assignment of a sample that should be assigned to bin B (0.1%-0.9%) to bin C (≥ 1%). This will result in the sample being cleaned up now, rather than being held for further consideration. This type of error is of low concern, because it is considered probable that many samples in bin B may ultimately require some sort of remedial action, so taking action sooner rather than later is not a serious penalty.

Case 2. The second type of mis-classification is assignment of a sample that should be assigned to bin A (<0.1%) to bin B (0.1%-9%). Because no action is currently scheduled for samples in bin B, this mis-assignment does not result in any immediate consequence. Further, it is expected that more data will be collected before final decisions are made on how to proceed with samples in bin B. Thus, this type of mis-classification is also of low concern, because the new data would likely result in the correct classification of the sample before action is taken.

False Negatives

Case 3. The third type of mis-classification is assignment of a sample that should be in bin C ($\geq$ 1%) to bin B (0.1%-0.9%). This is not of major concern, because samples in bin B are not considered to be "clean" and all of the samples in bin B will be evaluated further before final decision making. Thus, the mis-classification may be corrected either by improved analysis results, or the sample may be remediated in any event.

Case 4. The fourth type of mis-classification is assignment of a sample in bin B (0-1-0.9%) to bin A (<0.1%). This mis-classification is of concern, since samples in bin A are currently given the lowest priority and ultimately it could be decided that no action is warranted for such samples. Thus, assignment of a bin B sample to bin A is the most important error to avoid.

Within this type of error class, the degree of concern depends on where in bin B the true value lies. For example, it would be extremely undesirable to assign a sample whose true concentration was 0.8% to bin A, while the consequences of assigning a sample whose true concentration is 0.12% to bin A is not as severe. For convenience, assignment of a sample whose true concentration is 0.3% or above to bin A will be referred to as a Case 4a error, while assignment of a samples whose true concentration is 0.1%-0.3% will be referred to as a Case 4b error.

Results Based on the Semi-Quantitative Approach

SEM Results

Table 3 presents the results of the application of the semi-quantitative method to the SEM data from EMSL and RESI. As seen, if the combined set of all USGS spiked samples had been assigned to bins based on SEM, assignment would have been correct in 50%-77% of the cases. Most of the discordant samples were those that contained the fine-grained spiking material in DFC soils, with 11%-44% of these samples being correctly assigned and the remainder (all in bin B) being reported as non-detects. If this set of samples is judged to be atypical (because the fine ground fibers are not likely to be the main component of field samples with concentrations above 0.1% by mass) and is excluded from the evaluation, the assignment to bins would have been 71%-94% accurate.

The mis-classification rates for the USGS-spiked ISTM samples analyzed by SEM are summarized below:

Laboratory	Matrix	Amphibole	Correct	Case 1 B->C	Case 2 A->B	Case 3 C->B	Case 4a High B->A	Case 4b Low B->A
EMSL	DFC	Coarse	88%	0%	0%	0%	0%	13%
	DFC	Fine	44%	0%	0%	11%	0%	44%
	Libby	Coarse	100%	0%	0%	0%	0%	0%
	DFC+Libby	Coarse	94%	0%	0%	0%	0%	6%
RESI	DFC	Coarse	63%	13%	0%	0%	0%	25%
	DFC	Fine	11%	0%	0%	0%	44%	44%
	Libby	Coarse	78%	0%	0%	0%	11%	11%
	DFC+Libby	Coarse	71%	6%	0%	0%	6%	18%

As seen, the false positive rate (Case 1 and Case 2) is low, and if the DFC Fine grained samples are excluded, false negatives (Case 3 and Case 4) are mainly of the Case 4b type (this is of lesser concern than Case 4a).

The last entry in Table 3 shows the degree of concordance for samples whose concentration was quantifiable ($\geq$ 1%) by PLM (this corresponds to bin C). As seen, concordance was low in this case (0%-40%), with SEM tending to provide a lower concentration estimate than PLM. As noted above, it is not known whether this is because SEM is tending to underestimate and/or because PLM is tending to overestimate true concentrations.

Concordance by IR

Table 4 presents the semi-quantitative results for samples analyzed by IR. As seen, if the USGS spiked samples had been assigned to bins based on IR, assignment would have been correct in 62% of the cases. However, most of the errors were due to the inability to detect the fine-grained spiking material in DFC soils (except in the sample spiked at 2%). If this set of samples is excluded, the assignment to bins would have been 88% accurate.

The mis-classification rates for the USGS-spiked ISTM samples analyzed by IR are summarized below:

Matrix	Amphibole	Correct	Case 1 B->C	Case 2 A->B	Case 3 C->B	Case 4a High B->A	Case 4b Low B->A
DFC	Coarse	88%	0%	0%	0%	0%	13%
DFC	Fine	11%	0%	0%	0%	44%	44%
Libby	Coarse	89%	0%	0%	0%	0%	11%
DFC+Libby	Coarse	88%	0%	0%	0%	0%	12%

As seen, the false positive rate (Case 1 and Case 2) is zero, and if the DFC Fine grained samples are excluded, false negatives (Case 3 and Case 4) are of the Case 4b type (this is of lesser concern than Case 4a).

The last entry in Table 4 shows the degree of concordance for samples whose concentration was quantifiable ($\geq$ 1%) by PLM (this corresponds to bin C). As seen, concordance was about 60%, with IR classifying 3 of 5 samples as being at or above 1%.

5.0 INTERIM CONCLUSION

Based on these initial results, it is concluded that even though neither of the current methods (SEM or IR) for analysis of asbestos in soil appears to have high accuracy, both offer the potential of being useful as semi-quantitative screening tool for assessment of soil samples. Because IR is faster and less costly than SEM, this approach will be used to begin analysis of site samples, along with parallel analysis of some soil samples by SEM, PLM and other methods, as needed to further refine the technique.

6.0 FOLLOW-UP STUDY

The results of the current pilot study are limited by a number of factors, including the following:

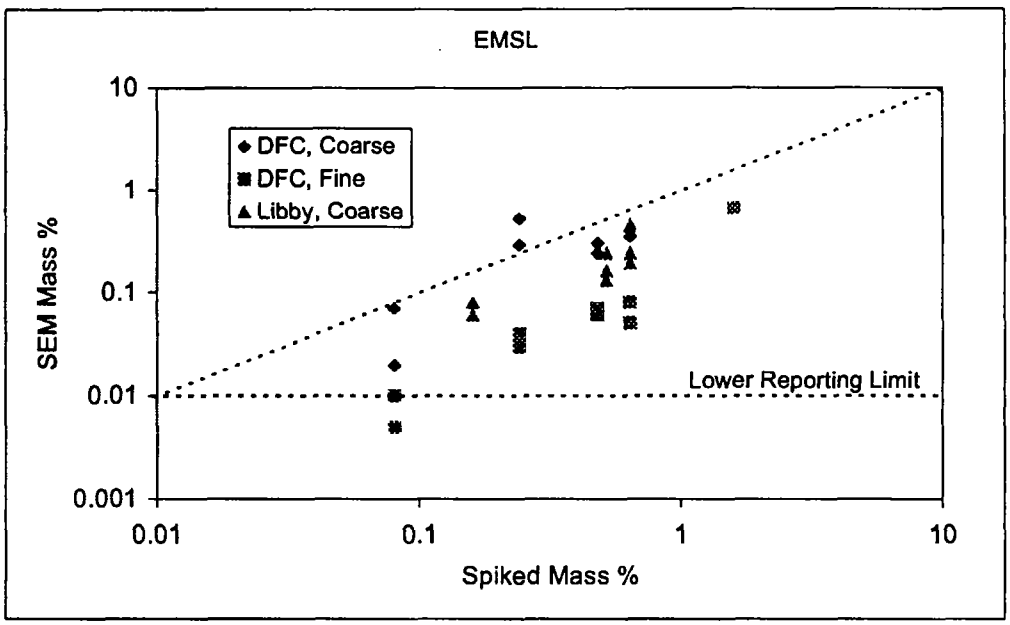
- The range of asbestos concentrations in the test materials spanned a relatively narrow range, limiting the ability to assess the performance of the methods. In particular, with regard to tests on semi-quantitative binning success, 25 out of 26 of the USGS-spiked samples were within bin B, with one sample in bin C. There were five Libby site soils ranked by PLM as having quantifiable concentration values of 1% or above, but since the PLM results themselves are uncertain, it is not known how many of these samples were authentic bin C samples.

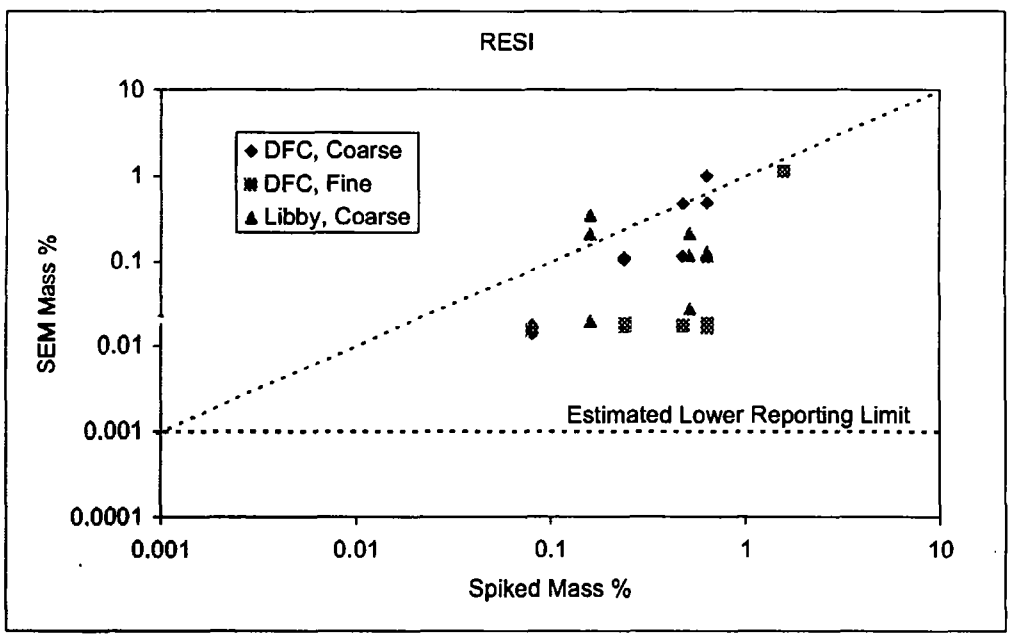
- No unspiked samples of DFC soil or prepared Libby soil were included, preventing a clear determination of the lowest levels that can be distinguished from background by each method.

- The Libby soil used to prepare spiked samples was prepared by mechanical disaggregation and sieveing through a 2 mm screen, procedures that are not currently applied to Libby field soil samples. As a consequence, the soil matrix is not characteristic of what is observed in field soils. Indeed, both analytical laboratories reported the soil matrix in these samples was un-representative of authentic Libby field samples.

For these reasons, additional testing is needed to more fully evaluate the relative accuracy and precision of IR, SEM and PLM. A proposed study design for such a study is presented separately.

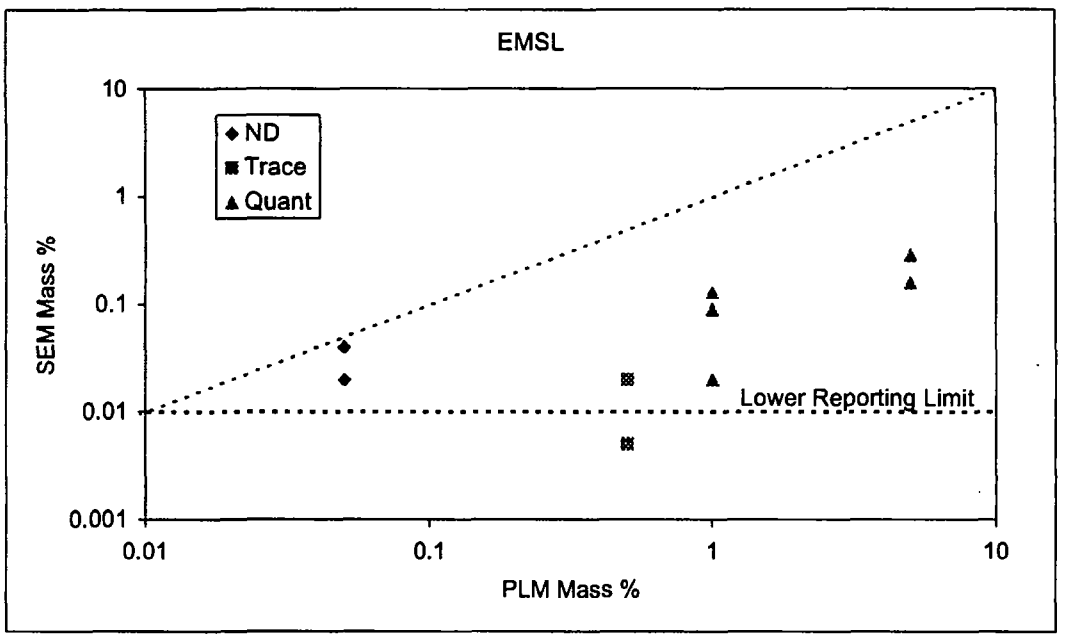
FIGURE 1. SEM RESULTS FOR USGS SPIKED ISTM SAMPLES

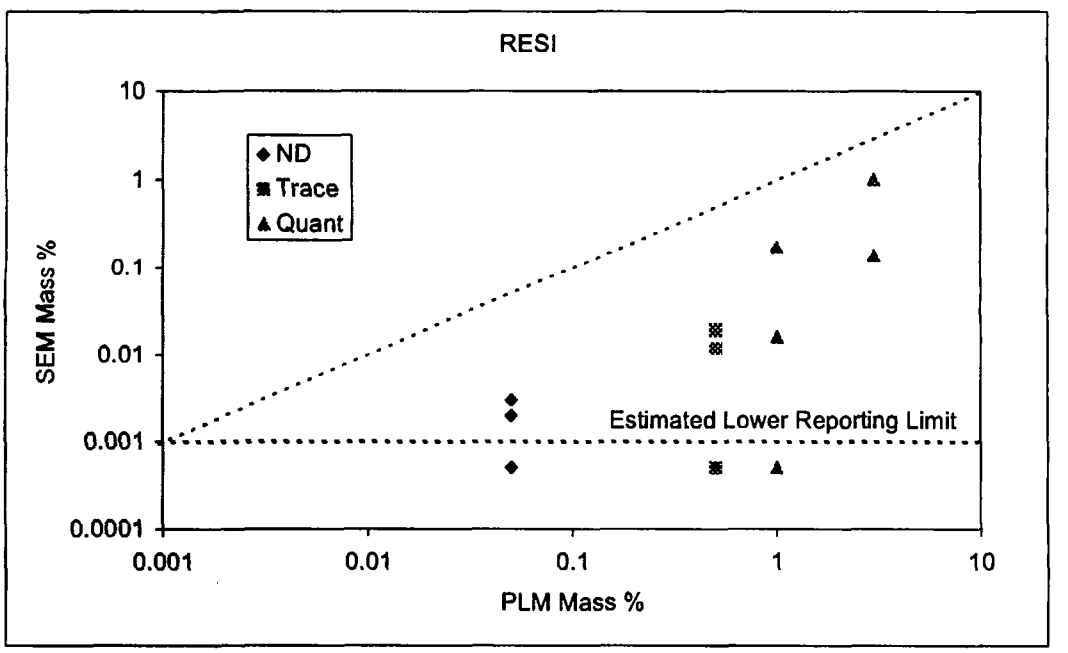




NOTE: Non-detect values are indicated by points slightly below the reporting limit.

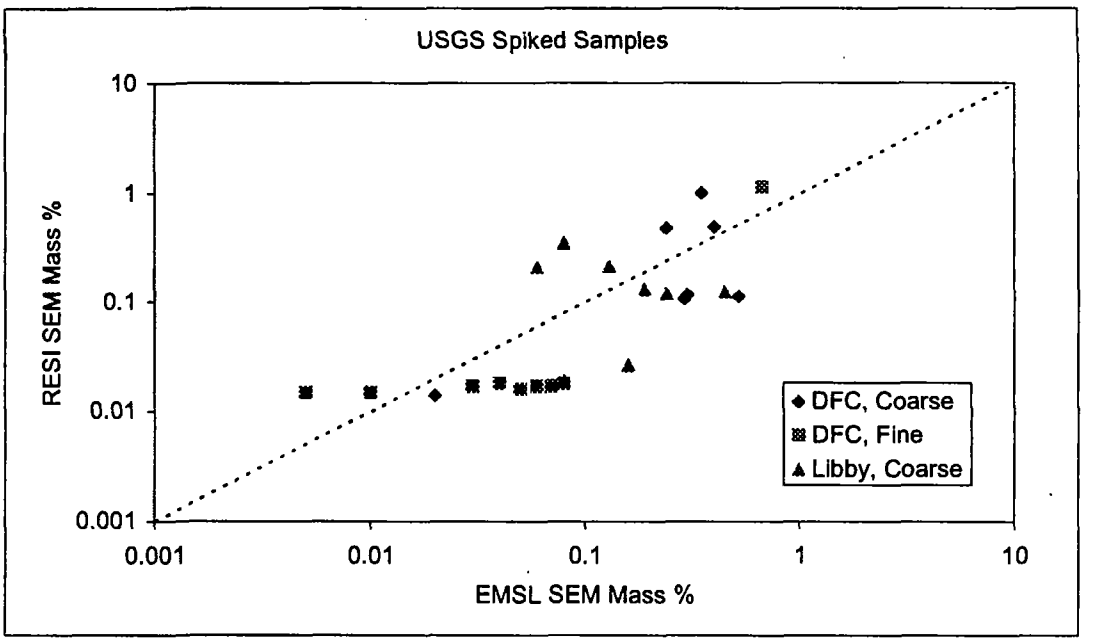
FIGURE 2. SEM RESULTS FOR LIBBY FIELD SAMPLES





NOTE: Non-detect values are indicated by points slightly below the reporting limit.

FIGURE 3. INTERLAB COMPARISON OF SEM RESULTS



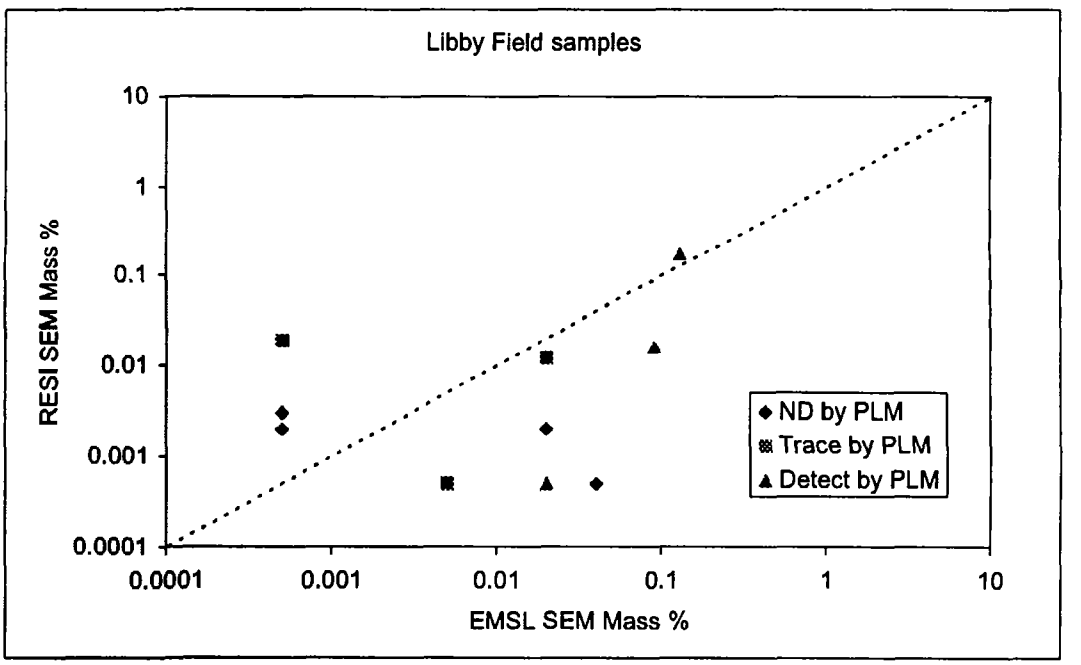
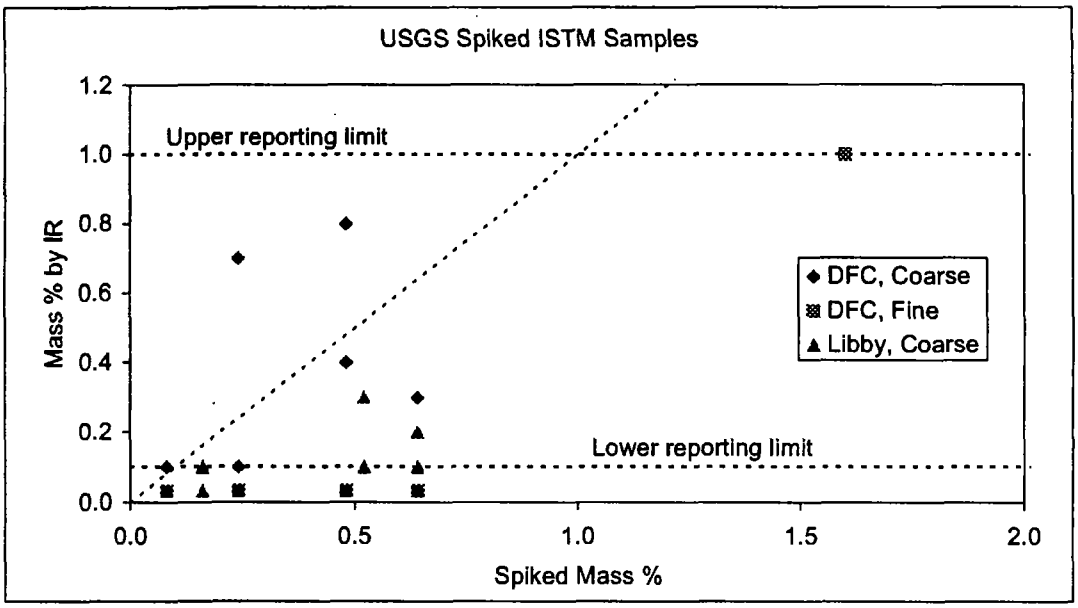
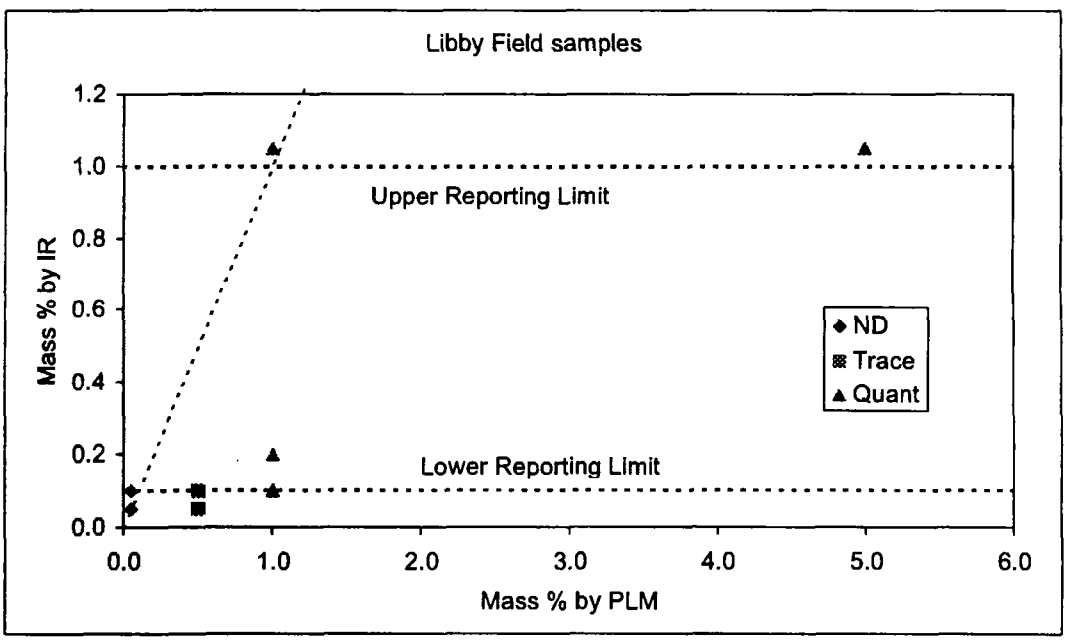


FIGURE 4. IR RESULTS (EMSL)





NOTE: Non-detect values are indicated by points slightly below the reporting limit.

TABLE 1. SUMMARY OF ISTM SAMPLES

USGS ID Number	Libby Number	Soil Type	Spike material	PLM Conc	Spiked Mass %		Sent To	
					Total	Asbestos	EMSL	RESI
GSCD0A11		DFC	Coarse		0.1	0.08	x	x
GSCD0A60		DFC	Coarse		0.6	0.48	x	x
GSCD0B10		DFC	Coarse		0.1	0.08	x	x
GSCD0B32		DFC	Coarse		0.3	0.24	x	x
GSCD0C31		DFC	Coarse		0.3	0.24	x	x
GSCD0D82		DFC	Coarse		0.8	0.64	x	x
GSCD0F61		DFC	Coarse		0.6	0.48	x	x
GSCD0F81		DFC	Coarse		0.8	0.64	x	x
GSFD0011		DFC	Fine		0.1	0.08	x	x
GSFD0012		DFC	Fine		0.1	0.08	x	x
GSFD0031		DFC	Fine		0.3	0.24	x	x
GSFD0032		DFC	Fine		0.3	0.24	x	x
GSFD0060		DFC	Fine		0.6	0.48	x	x
GSFD0061		DFC	Fine		0.6	0.48	x	x
GSFD0081		DFC	Fine		0.8	0.64	x	x
GSFD0082		DFC	Fine		0.8	0.64	x	x
GSFDD02		DFC	Fine (dry mix)		2	1.6		x
GSFDDA2		DFC	Fine (dry mix)		2	1.6	x	
GSCL0A20		Libby bkg (sieved)	Coarse		0.2	0.16	x	x
GSCL0A80		Libby bkg (sieved)	Coarse		0.8	0.64	x	x
GSCL0A81		Libby bkg (sieved)	Coarse		0.8	0.64	x	x
GSCL0B22		Libby bkg (sieved)	Coarse		0.2	0.16	x	x
GSCL0C66		Libby bkg (sieved)	Coarse		0.65	0.52	x	x
GSCL0D65		Libby bkg (sieved)	Coarse		0.65	0.52	x	x
GSCL288		Libby bkg (sieved)	Coarse		0.8	0.64	x	x
GSCL465		Libby bkg (sieved)	Coarse		0.65	0.52	x	x
GSCL802		Libby bkg (sieved)	Coarse		0.2	0.16	x	x
GSS0943C	1-00943	libby soil #0943	None	ND			x	x
GSSA00108	A00108	libby soil #108	None	ND			x	x
GSSA00112	A00112	libby soil #112	None	ND			x	x
GSS103813	1-03813	libby soil #3813	None	ND			x	x
GSSA00107	A00107	libby soil #107	None	Trace			x	x
GSSA00110	A00110	libby soil #110	None	Trace			x	x
GSS103806	1-03806	libby soil #3806	None	Trace			x	x
GSS0942C	1-00942	libby soil #0942	None	1%			x	x
GSSA00109	A00109	libby soil #109	None	1%			x	x
GSS103808	1-03808	libby soil #3808	None	1%			x	x
GSDM001	1-04152	Libby Soil (CDM)	None	3%				x
GSDM002	1-04152	Libby Soil (CDM)	None	3%				x
GSDM003	1-03407	Libby Soil (CDM)	None	5%			x	
GSDM004	1-03407	Libby Soil (CDM)	None	5%			x	

TABLE 2. RESULTS

USGS ID Number	Soil Type	Spike material	Nominal Mass %		SEM Results		IR Results
			PLM	Spiked	EMSL	RESI	EMSL
GSCD0A11	DFC	Coarse		0.08	0.02	0.014	< 0.1
GSCD0A60	DFC	Coarse		0.48	0.24	0.48	0.8
GSCD0B10	DFC	Coarse		0.08	0.07	0.017	0.1
GSCD0B32	DFC	Coarse		0.24	0.29	0.107	0.1
GSCD0C31	DFC	Coarse		0.24	0.52	0.113	0.7
GSCD0D82	DFC	Coarse		0.64	0.35	1.019	0.3
GSCD0F61	DFC	Coarse		0.48	0.3	0.117	0.4
GSCD0F81	DFC	Coarse		0.64	0.4	0.49	0.3
GSFD0011	DFC	Fine		0.08	0.01	0.015	< 0.1
GSFD0012	DFC	Fine		0.08	< 0.01	0.015	< 0.1
GSFD0031	DFC	Fine		0.24	0.03	0.017	< 0.1
GSFD0032	DFC	Fine		0.24	0.04	0.018	< 0.1
GSFD0060	DFC	Fine		0.48	0.07	0.017	< 0.1
GSFD0061	DFC	Fine		0.48	0.06	0.017	< 0.1
GSFD0081	DFC	Fine		0.64	0.08	0.018	< 0.1
GSFD0082	DFC	Fine		0.64	0.05	0.016	< 0.1
GSFDD02	DFC	Fine (dry mix)		1.6		1.152	
GSFDDA2	DFC	Fine (dry mix)		1.6	0.67		> 1
GSCL0A20	Libby bkg (sieved)	Coarse		0.16	0.08	0.35	< 0.1
GSCL0A80	Libby bkg (sieved)	Coarse		0.64	0.19	0.13	0.2
GSCL0A81	Libby bkg (sieved)	Coarse		0.64	0.45	0.123	0.1
GSCL0B22	Libby bkg (sieved)	Coarse		0.16	0.06	0.21	0.1
GSCL0C66	Libby bkg (sieved)	Coarse		0.52	0.16	0.0265	0.1
GSCL0D65	Libby bkg (sieved)	Coarse		0.52	0.13	0.214	0.3
GSCL288	Libby bkg (sieved)	Coarse		0.64	0.24	0.118	0.1
GSCL465	Libby bkg (sieved)	Coarse		0.52	0.24	0.119	0.1
GSCL802	Libby bkg (sieved)	Coarse		0.16	0.08	0.019	0.1
GSS0943C	libby soil #0943	None	ND		0.04	ND	0.1
GSSA00108	libby soil #108	None	ND		ND	0.003	0.1
GSSA00112	libby soil #112	None	ND		ND	0.002	0.1
GSS103813	libby soil #3813	None	ND		0.02	0.002	< 0.1
GSSA00107	libby soil #107	None	Trace		< 0.01	ND	0.1
GSSA00110	libby soil #110	None	Trace		0.02	0.012	< 0.1
GSS103806	libby soil #3806	None	Trace		ND	0.019	< 0.1
GSS0942C	libby soil #0942	None	1%		0.13	0.176	> 1
GSSA00109	libby soil #109	None	1%		0.09	0.016	0.2
GSS103808	libby soil #3808	None	1%		0.02	ND	0.1
GSDM001	Libby Soil (CDM)	None	3%			1.024	
GSDM002	Libby Soil (CDM)	None	3%			0.139	
GSDM003	Libby Soil (CDM)	None	5%		0.29		> 1
GSDM004	Libby Soil (CDM)	None	5%		0.16		> 1

TABLE 3. CONCORDANCE FOR SEM

DFC Coarse	Nominal	EMSL			RESI		
		Bin A	Bin B	Bin C	Bin A	Bin B	Bin C
Bin A	0						
Bin B	8	1	7		2	5	1
Bin C	0						
Concordance		88%			63%		

DFC Fine	Nominal	EMSL SEM			RESI		
		Bin A	Bin B	Bin C	Bin A	Bin B	Bin C
Bin A	0						
Bin B	8	4	4		8		
Bin C	1		1				1
Concordance		44%			11%		

Libby Coarse	Nominal	EMSL			RESI		
		Bin A	Bin B	Bin C	Bin A	Bin B	Bin C
Bin A	0						
Bin B	9		9		2	7	
Bin C	0						
Concordance		100%			78%		

All Spiked samples	Nominal	EMSL			RESI		
		Bin A	Bin B	Bin C	Bin A	Bin B	Bin C
Bin A	0	0	0	0	0	0	0
Bin B	25	5	20	0	12	12	1
Bin C	1	0	1	0	0	0	1
Concordance		77%			50%		

All except DFC Fine	Nominal	EMSL			RESI		
		Bin A	Bin B	Bin C	Bin A	Bin B	Bin C
Bin A	0	0	0	0	0	0	0
Bin B	17	1	16	0	4	12	1
Bin C	0	0	0	0	0	0	0
Concordance		94%			71%		

Field Samples Unspiked	Nominal	EMSL			RESI		
		Bin A	Bin B	Bin C	Bin A	Bin B	Bin C
Bin C	5	1	4		1	2	2
Concordance		0%			40%		

Bins

A = less than 0.1%
B = 0.1% to 0.9%
C = greater than or equal to 1%

TABLE 4. CONCORDANCE FOR IR

DFC Coarse	Nominal	EMSL IR Bin A	Bin B	Bin C
Bin A	0			
Bin B	8	1	7	
Bin C	0			
Concordance		88%		

DFC Fine	Nominal	EMSL IR Bin A	Bin B	Bin C
Bin A	0			
Bin B	8	8		
Bin C	1			1
Concordance		11%		

Libby Coarse	Nominal	EMSL IR Bin A	Bin B	Bin C
Bin A	0			
Bin B	9	1	8	
Bin C	0			
Concordance		89%		

All Spiked samples	Nominal	EMSL IR Bin A	Bin B	Bin C
Bin A	0			
Bin B	25	10	15	
Bin C	1			1
Concordance		62%		

All except DFC Fine	Nominal	EMSL IR Bin A	Bin B	Bin C
Bin A	0	0	0	0
Bin B	17	2	15	0
Bin C	0	0	0	0
Concordance		88%		

Field Samples Unspiked	Nominal	EMSL IR Bin A	Bin B	Bin C
Bin C	5		2	3
Concordance		60%		

Bins

A = less than 0.1%
B = 0.1% to 0.9%
C = greater than or equal to 1%